Gamma-Titanium Intermetallic Alloy produced by Selective Laser Melting using Mechanically Alloyed and Plasma Spheroidized Powders

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Abstract Conventional manufacturing of titanium intermetallic alloys is associated with brittleness, hard machinability and, consequently, the high cost, which makes additive manufacturing a promising way of producing complex intermetallic parts. At the same time, γ-TiAl alloys exhibit good high temperature strength, fatigue and oxidation resistance. In the present study the gamma-based alloy spherical powders were prepared by mechanical alloying from elemental powders followed by the plasma spheroidization process. Microstructure and phase composition of the produced powders were studied after different milling times in a planetary mill. The optimally milled powders were treated in the flow of a thermal plasma to obtain powder particles with a high degree of sphericity. The produced spherical powders were used in Selective Laser Melting (SLM) process with high preheating temperatures to obtain crack-free intermetallic samples. The microstructure and phase composition of the SLM-ed TiAl-samples were investigated with regard to different process parameters.

Keywords: mechanical alloying, plasma spheroidization, titanium alloy, selective laser melting, additive manufacturing

1. Introduction

Intermetallic titanium aluminide γ-TiAl-based alloys are promising materials for manufacturing light-weight components of aerospace and automotive engines due to their high strength at elevated temperatures, low density and good oxidation and creep resistance [1,2]. Titanium aluminide alloys are considered to be candidates for replacing nickel-based superalloys, which are used for high-temperature parts in turbine engines [3]. However, TiAl-based alloys possess very low room temperature ductility and poor hot deformability and are prone to cracking during conventional processing methods. This makes manufacturing parts from TiAl-based alloys very cost and time-consuming. Existing conventional ways, e.g. extrusion or casting and isothermal forging, have extremely high costs and results in heterogeneous microstructure. Thus, processing TiAl-based alloys requires further development, e.g. application of powder metallurgy and Additive Manufacturing (AM) processing [4].

AM technologies, such as Selective Laser Melting (SLM) and Electron Beam Melting (EBM), allow the fabrication of complex-shaped components with high mechanical properties from powder feedstock [5]. While AM of titanium aluminide components is a promising way of producing complex parts, the availability of TiAl-based powders suitable for AM processes is still limited. Mechanical alloying (MA) allows obtaining powders with complex chemical composition, however the particles obtained by MA have irregular shape and usually require post-processing to be used in AM. Plasma spheroidization (PS) process can be used to give the powder particles spherical shape [6]. During plasma treatment at high temperatures, the particles are rapidly melted and then solidified resulting in a homogeneous microstructure and spherical shape [7].

Ti-48Al-2Cr-2Nb (at. %) is one of the most widely use TiAl-based alloys. Addition of Nb to the alloy composition improves its high temperature resistance and addition of Cr increases its oxidation resistance [8].

In the current work, Ti-48Al-2Cr-2Nb intermetallic alloy powders were fabricated using elemental powders by MA followed by PS process. The microstructure, phase composition, and chemical composition of the produced powders after MA and PS were investigated. It was demonstrated that MA followed by PS can be used to fabricate spherical powders of TiAl-based alloy with homogeneous microstructure and composition. The obtained TiAl-powders were used in the SLM process to fabricate crack-free bulk samples with high-temperature pre-heating.

2. Materials and Methods

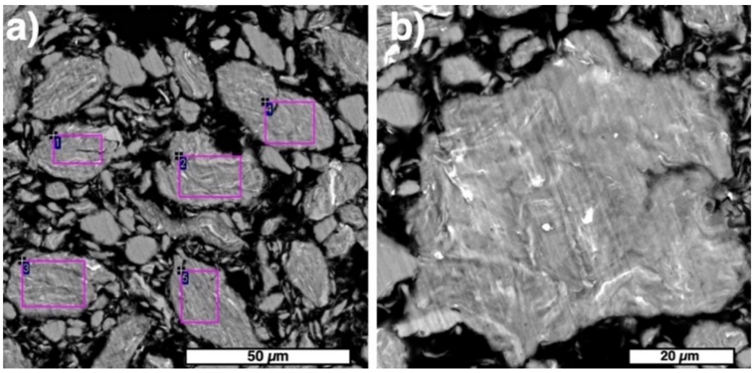
The elemental powders of Ti, Al, Cr, and Nb (with 99.9 % purity) were mixed to obtain the composition of Ti-48Al-2Cr-2Nb (at. %) and then mechanically alloyed using a planetary mill Fritsch Pulverisette 4 in the argon atmosphere. The milling of the powders was stopped every 15 min for 15 min to prevent overheating of the powders with the change of rotating direction for the main disk and the bowls. The total time of the MA process was 10 hours. Steel balls of 12 mm diameter were used as the milling media with the ball-to-powder ratio of 15:1. The main disk rotation speed was 300 r/min (clockwise direction) and the bowl rotation speed was 600 r/min (counterclockwise direction). After mechanical alloying the obtained powders were sieved using a 125 µm sieve and then spheroidized in the Tekna TEK-15 plant with the inductively coupled plasma at 15 kW plasma torch power. The Ar-He gas was used as the plasma forming gas. The powder feeding rate was set to 15 g/min.

AconityMIDI SLM system was used to fabricate bulk samples from spherical powders. The system is equipped with 1000 W fiber laser with 1060 nm wavelength. An inductive heating element was utilized to preheat a titanium base plate to 800 ºC. The laser power, layer thickness and hatching distance were set correspondingly to 140 W, 30 µm and 110 µm, while the scanning speed were varied from 650 to 1250 mm/s to produce cylindrical samples with 10 mm diameter and 10 mm height at volume energy densities varying from 36 to 70 J/mm^3. During the SLM process, the process chamber was purged with Argon gas. The laser spot diameter was kept at around 80 µm. The densities of the samples were measured using the Archimedes' method.

TESCAN Mira 3 LMU scanning electron microscope (SEM) with secondary electrons (SE) and backscattered electrons (BSE) was utilized for the microstructural characterization. Energy-dispersive X-ray spectroscopy (EDS) option was used for local chemical analysis of the samples. The phase composition was analyzed with a Bruker D8 Advance X-ray diffraction (XRD) meter using Cu-Kα (λ = 1.5418 Å) irradiation.

3. Results and Discussion

Fig. 1 shows the BSE-SEM images of Ti-48Al-2Cr-2Nb powder particles' cross-sections after 10 h of mechanical alloying (MA). The powders obtained by MA have irregular particles' shape, which is typical for the powders fabricated by milling processes. From BSE-SEM images it can be seen that the particles feature inhomogeneous microstructure and distribution of chemical elements. There are white areas corresponding to Nb and Cr elements. In general, the particles after MA have layered type of microstructure typical for mechanically alloyed powders [9-11]. The EDS-maps (Fig. 2) of the alloying elements in the powder particles obtained by MA show that Ti and Al are distributed relatively homogeneously, while Nb and Cr are characterized by areas with uneven distribution. The chemical composition of the powders according to the EDS results (Table 1) is in accordance with the initial composition of the elemental powder blend. However, there is a small Fe contamination of the powders after MA due to the use of steel grinding media. According to the XRD results (Fig. 3), the powder after MA consists of hexagonal closed-packed titanium phase and aluminum. There are also small peaks corresponding to α_2-Ti_3Al phase.



Area	Element content, at. %				
	Ti	Al	Nb	Cr	Fe
1	49.4	46.5	2.2	1.9	–
2	51.2	45.3	1.9	1.6	–
3	48.8	48.1	4.4	1.2	–
4	48.8	47.6	2.0	1.5	0.14
5	49.0	47.9	1.5	1.9	0.08
Mean	49.4	47.1	1.9	1.5	0.04

Figure 1 BSE-SEM images of Ti-48Al-2Cr-2Nb powders particles cross-sections after MA for 10 h at different magnification. The areas used for the EDS analysis reported in the table are shown in a)

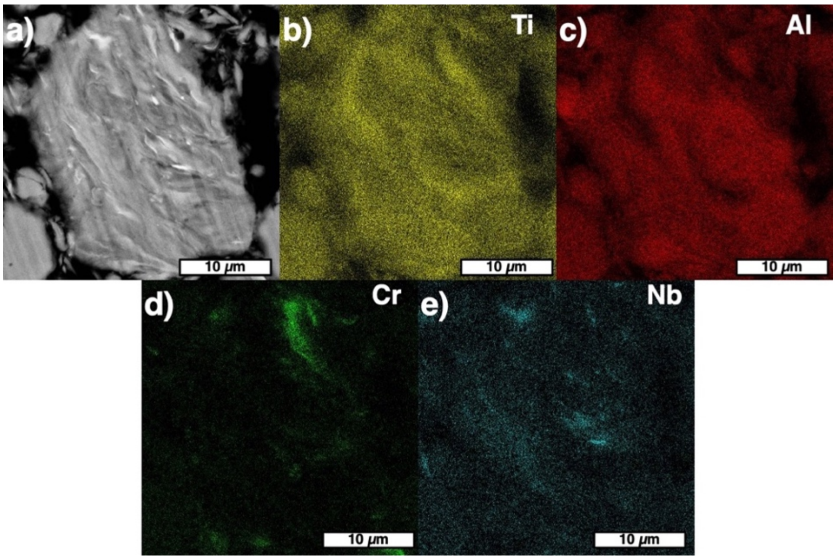


Figure 2 BSE-SEM image of the Ti-48Al-2Cr-2Nb particle's polished cross-section for the powder after MA and elements distribution

After the treatment of the mechanically alloyed powders in the high-temperature plasma jet, the resulted particles are characterized by spherical shape (Fig. 4). The XRD results show that after PS the powder consists of Ti_3Al-phase and TiAl-phase, while Al and Ti peaks disappeared. The main phase is Ti_3Al. According to the EDS-analysis, the fabricated powder has the following chemical composition: Ti-40.4Al-2.1Cr-2.5Nb-0.3Fe (at. %). The Al content decreased by 7 % (at.) after plasma spheroidization compared to the mechanical alloyed powder. This might be the result of partial evaporation of Al during plasma treatment. The reduced Al content might be not sufficient enough to form sufficient amount of TiAl-phase [12, 13]. Thus, it is needed to increase Al content in the initial powder blend to obtain an alloy of the required composition. The cross-section of the fabricated powder particle shows dendritic microstructure [14] and consists mainly of supersaturated α_2-Ti_3Al phase (Fig. 5, a).

The final particle size distribution of the powder after sieving used in the SLM process was the following: d_{10} = 9.7 µm, d_{50} = 33.3 µm, d_{10} = 67.7 µm.

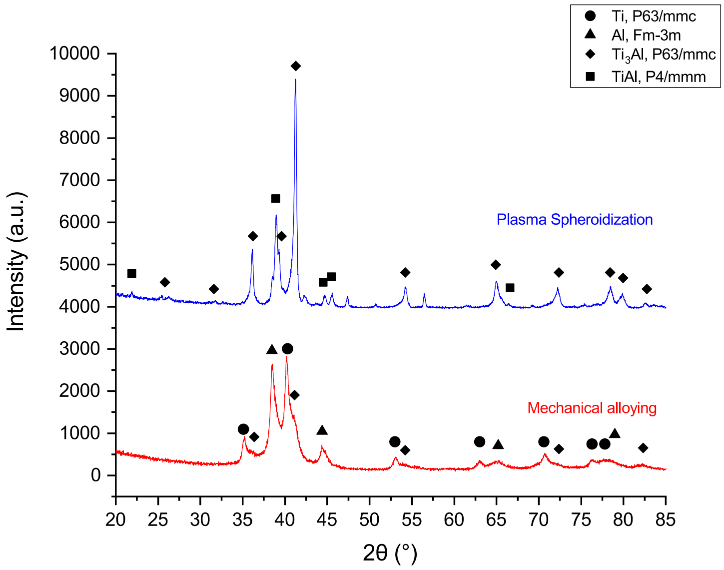


Figure 3 The XRD results of the powders after mechanical alloying and after plasma spheroidization processes

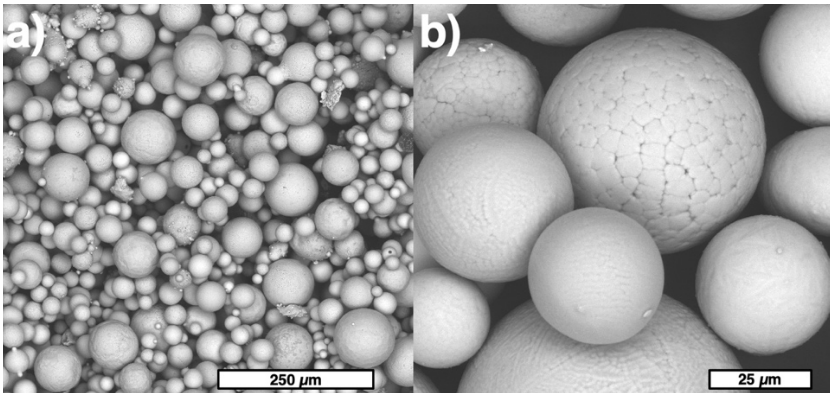


Figure 4 SEM-images of Ti-48Al-2Cr-2Nb powder particles after plasma spheroidization

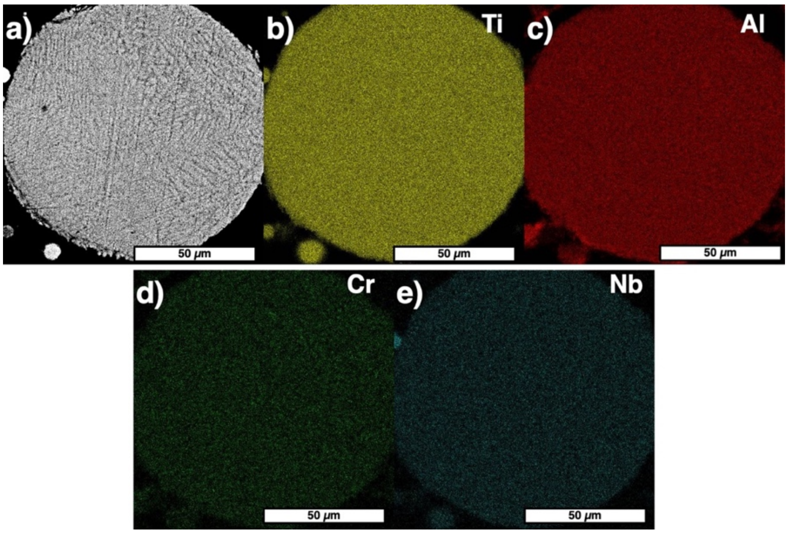


Figure 5 EDS-maps of Ti-48Al-2Cr-2Nb powders particle cross-sections after plasma spheroidization

The photo of the fabricated TiAl-samples is shown in Fig. 6, a. The highest density of 4.16 ± 0.05 g/cm^3 was achieved at 800 mm/s scanning speed (Fig. 6, b), which corresponds to energy density value of 57 J/mm^3. Further increasing scanning speed leads to decrease in density of the samples. The achieved values show that the produced samples have relatively high densities since the typical density for Ti48Al2Cr2Nb alloy is about 3.9–4.1 g/cm^3. The chemical composition of the TiAl-sample measured by EDX is the following (in at. %): Al – 41.8 %, Cr – 2.2 %, Nb –2.2 %, Fe – 0.4 %, Ti – balance. This indicated that there is about 2% Al loss during the SLM process compared to the starting powder after PS.

(a)

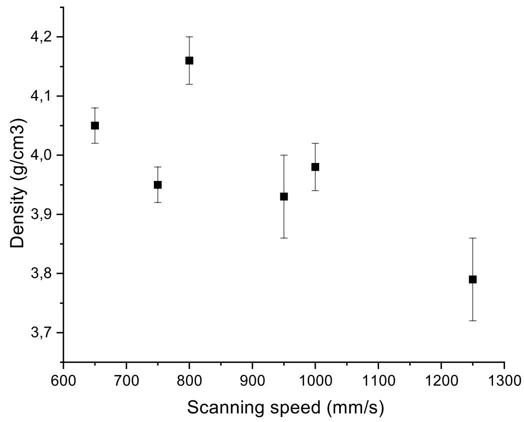
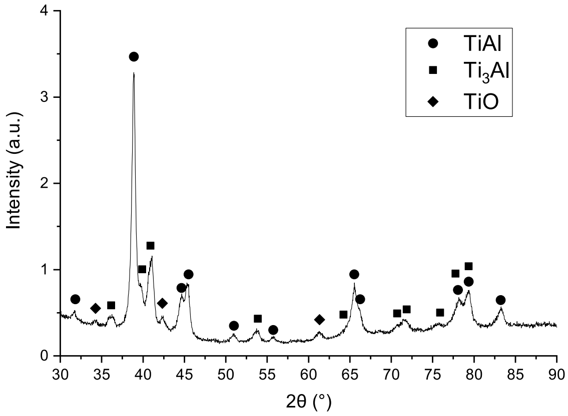

(b)


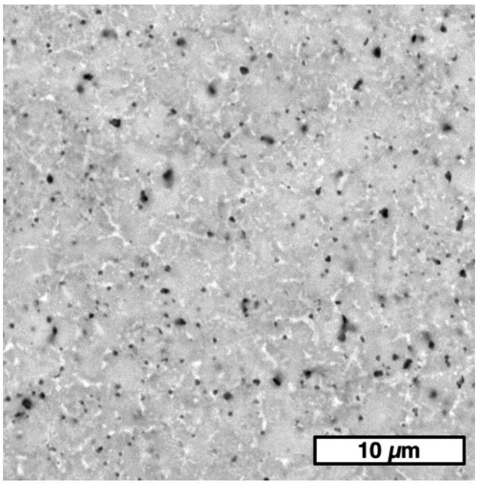
Figure 6 The fabricated TiAl-samples (a) and their densities depending on scanning speed values

According to the XRD results (Fig. 7, a), the produced samples consist mainly of γ-TiAl phase and α_2-Ti_3Al phase. Small amount of TiO peaks is also present on the XRD pattern, which indicates the presence of oxides formed either as a reaction between Ti and O from the starting powder or during the presence of oxygen in the process chamber atmosphere during the SLM process or cooling from the preheated state.



(a)

(b)



(c)

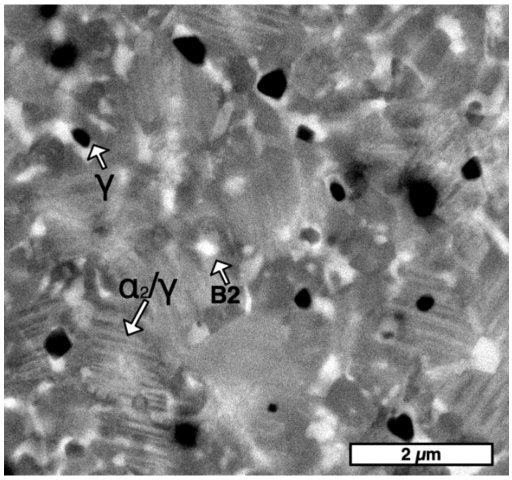


Figure 7 The XRD pattern (a) and SEM-BSE images of the microstructure of the as-fabricated Ti-48Al-2Cr-2Nb alloy sample at different magnification (b, c)

Fig. 7 (b, c) shows SEM-images in backscattered electrons (BSE) mode of the TiAl-samples produced by SLM in the as-built state. The microstructure features fine grains of α_2/γ-phases surrounded by Nb-enriched B2-phase (shown in white) as shown in Fig. 7, b). Fine globular β/B2 and γ-grains are present in the sample. Lamellar α_2/γ-phase areas can be seen at high magnification (Fig. 7, c). Similar microstructure was found in the TiAl-alloy obtained by SLM from gas atomized powders [15].

4. Conclusions

In this study, titanium aluminide alloy spherical powder was fabricated by the combination of mechanical alloying and plasma spheroidization processes from Ti-48Al-2Cr-2Nb elemental powder blend. The resultant chemical composition of the powder was Ti-40.4Al-2.1Cr-2.5Nb-0.3Fe (at. %), which is characterized by a decreased Al content due to its partial evaporation during plasma treatment and a small Fe contamination from steel grinding media. While the mechanical alloying resulted in powder particles with high degree of chemical inhomogeneity and irregular particle shape, the following plasma spheroidization process allowed obtaining highly spherical powder particles with homogeneous distribution of alloying elements. The produced powder consists of Ti_3Al as the main phase and TiAl-phase and is characterized by dendritic structure.

The obtained spherical powders were used in the SLM process with high-temperature preheating to fabricate crack-free samples. The highest density of 4.16 g/cm^3 was achieved for the sample fabricated at 57 J/mm^3 energy density. The phase composition of the samples consists mainly of γ-TiAl phase and α_2-Ti_3Al phase, as well as, small amount of TiO. The microstructure is characterized by α_2/γ-lamellar microstructure surrounded by Nb-enriched B2-phase along with fine globular γ-grains.

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